

# **Journal of Magnetic Resonance**

---

**EDITOR:** Wallace S. Brey, Jr.

**EDITORIAL BOARD:**

E. Raymond Andrew  
Edwin D. Becker  
James W. Cooper  
B. P. Dailey  
P. Diehl  
Richard Ernst  
Ray Freeman  
J. H. Goldstein

David M. Grant  
R. K. Harris  
K. H. Haussler  
Charles S. Johnson, Jr.  
J. Jonas  
Lowell Kispert  
Masaji Kubo  
George C. Levy

Ralph Livingston  
Bruce McGarvey  
Rex E. Richards  
Max T. Rogers  
Thomas A. Scott  
Ian C. P. Smith  
J. B. Stothers  
Robert L. Vold

Volume 31, 1978



**ACADEMIC PRESS**  
**New York and London**

A Subsidiary of Harcourt Brace Jovanovich, Publishers

Copyright © 1978 by Academic Press, Inc.

ALL RIGHTS RESERVED

No part of this publication may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopy, recording, or any information storage and retrieval system, without permission in writing from the copyright owner.

The appearance of the code at the bottom of the first page of an article in this journal indicates the copyright owner's consent that copies of the article may be made for personal or internal use, or for the personal or internal use of specific clients. This consent is given on the condition, however, that the copier pay the stated per copy fee through the Copyright Clearance Center, Inc. (Operations Staff, P.O. Box 765, Schenectady, New York 12301), for copying beyond that permitted by Sections 107 or 108 of the U.S. Copyright Law. This consent does not extend to other kinds of copying, such as copying for general distribution, for advertising or promotional purposes, for creating new collective works, or for resale. Copy fees for pre-1978 articles are the same as those shown for current articles.



Made in Great Britain

## CONTENTS OF VOLUME 31

NUMBER 1, JULY 1978

SEYMOUR H. KOENIG. A Novel Derivation of the Solomon-Bloembergen-Morgan Equations: Application to Solvent Relaxation by Mn <sup>2+</sup> -Protein Complexes . . . . .	1
B. T. PENNINGTON AND J. R. CAVANAUGH. Lanthanide Ion Nuclear Magnetic Resonance Probe Studies of Benzoic Acids. The Agreement with an Axially Symmetric Model . . . . .	11
J.-P. MARCHAL AND D. CANET. NMR of Nitrogen-15 in Natural Abundance. Determination of Two- and Three-Bond Coupling Constants between Proton and Nitrogen-15, Using Difference Proton Fourier Transform Spectroscopy and Double Resonance . . . . .	23
E. HASLINGER AND R. M. LYNDEN-BELL. Investigation of the Internal Rotation of Methyl Groups by <i>T</i> <sub>1</sub> Relaxation Measurements . . . . .	33
K. ROSE AND R. G. BRYANT. Electrolyte Ion Correlation Times at Protein Binding Sites . . . . .	41
JEREMY K. M. SANDERS, CHRISTOPHER G. NEWTON, AND JOHN C. WATERTON. McConnell's <i>Q</i> in Metalloporphyrin Radical Cations . . . . .	49
PETER STILBS AND MICHAEL E. MOSELEY. Chemical Exchange Rates from Fourier Transform Measurements of Nuclear Spin-Lattice Relaxation in the Rotating Frame. Application to Hindered Internal Rotation in Ureas . . . . .	55
IAN J. COLQUHOUN AND WILLIAM MCFARLANE. Fourier Transform Heteronuclear Magnetic Triple Resonance in Complex Spin Systems: Tetramethylbiphosphine Disulfide . . . . .	63
JÜRGEN K. DOHRMANN AND WOLFGANG KIESLICH. Electron Spin Resonance Study of 1-Hydropyridinyl Radicals in Solution. 4-Acetylpyridine and Pyridine-4-carbaldehyde . . . . .	69
GEOFFREY BODENHAUSEN, RAY FREEMAN, GARETH A. MORRIS, AND DAVID L. TURNER. NMR Spectra of Some Simple Spin Systems Studied by Two-Dimensional Fourier Transformation of Spin Echoes . . . . .	75
J. L. DE WITT, M. A. HEMMINGA, AND T. J. SCHAAFSMA. A <sup>13</sup> C and <sup>1</sup> H NMR Study of the Dynamic Behavior of Tobacco Mosaic Virus Protein . . . . .	97
M. ZDANOWSKA, J. STANKOWSKI, AND M. MAĆKOWIAK. Pressure and Temperature Dependence of the Nuclear Quadrupole Resonance of <sup>35</sup> Cl in Chloracetamide . . . . .	109

JUKKA JOKISAARI, KAUKO RÄISÄNEN, LAURI LAJUNEN, AARNE PASSOJA, AND PEKKA PYYKKÖ. Proton, Carbon, and Cadmium NMR Measurements and Relativistic Calculation of the Cadmium–Carbon Coupling Tensor in Dimethyl Cadmium . . . . .	121
K. NAGAYAMA, P. BACHMANN, K. WÜTHRICH, AND R. R. ERNST. The Use of Cross-Sections and of Projections in Two-dimensional NMR Spectroscopy . . . . .	133
D. T. EDMONDS AND A. A. L. WHITE. Double Transitions in Deuterium Quadrupole Resonance . . . . .	149
<b>COMMUNICATIONS</b>	
A. STÁSKO, A. TKÁČ, AND Ľ. MALÍK. VII. An Unusual Doublet Splitting in the EPR Spectra of Anion Radicals in Hexamethylphosphortriamide Solution . . . . .	161
JOZEF KOWALEWSKI, ANDERS ERICSSON, AND RAGNAR VESTIN. Determination of NOE Factors Using the Dynamic Overhauser Enhancement Technique Combined with a Nonlinear Least-Squares-Fitting Procedure . . . . .	165
R. KAPTEIN, K. DIJKSTRA, F. MÜLLER, C. G. VAN SCHAGEN, AND A. J. W. G. VISSER. 360 MHz Laser-Induced Photo-CIDNP in Photoreactions of Flavins . . . . .	171

**NUMBER 2, AUGUST 1978**

TORBEN WAMSLER, JØRGEN TORMOD NIELSEN, ERIK JONAS PEDERSEN, AND KJELD SCHAUMBURG. NMR Studies of Pyridine-N-Oxide. Determination of Spectroscopic Constants from [ <sup>15</sup> N]-, [4- <sup>2</sup> H]-, and the Parent Species . . . . .	177
MARTIN D. BEER AND ROGER GRINTER. Calculations of the Nuclear Spin–Spin Coupling Constants of Second-Row Elements. II. One-Bond Couplings of Silicon and Phosphorus to Carbon . . . . .	187
CLAUDE R. LASSIGNE AND E. J. WELLS. Deuterium Isotope Effects in the <sup>1</sup> H, <sup>13</sup> C, and <sup>119</sup> Sn NMR Spectra for the Series Sn(CH <sub>3</sub> ) <sub>4-n</sub> (CD <sub>3</sub> ) <sub>n</sub> . . . . .	195
A. R. SIEDLE, G. M. BODNER, A. R. GARBER, R. F. WRIGHT, AND L. J. TODD. A High-Resolution <sup>11</sup> B NMR Study of the [(B <sub>10</sub> H <sub>12</sub> ) <sub>2</sub> Ni] <sup>2+</sup> Ion. . . . .	203
HOMMO T. EDZES AND EDWARD T. SAMULSKI. The Measurement of Cross-Relaxation Effects in the Proton NMR Spin–Lattice Relaxation of Water in Biological Systems: Hydrated Collagen and Muscle . . . . .	207
H. HANSSUM, W. MAURER, AND H. RÜTERJANS. Elimination of Systematic Errors in Fast Inversion–Recovery Spin–Lattice Relaxation Time Measurements . . . . .	231
Y. BEN TAARIT, J. C. VEDRINE, J. F. DUTEL, AND C. NACCACHE. EPR Investigation of the Structure and Reactivity of Pd(I) Species Generated in Synthetic Mordenite-Type Zeolite . . . . .	251

JACEK MICHALIK AND LARRY KEVAN. Temperature Dependence of Electron Spin-Lattice Relaxation of Radiation-Produced Silver Atoms in Polycrystalline Aqueous and Glassy Organic Matrices. Importance of Relaxation by Tunneling Modes in Disordered Matrices . . . . .	259
W. DAVID WILLIAMS, E. F. W. SEYMOUR, AND R. M. COTTS. A Pulsed-Gradient Multiple-Spin-Echo NMR Technique for Measuring Diffusion in the Presence of Background Magnetic Field Gradients . . . . .	271
A. C. McLAUGHLIN, C. GRATHWOHL, AND R. E. RICHARDS. The Interaction of Cobalt with Glycerophosphoryl Choline and Phosphatidyl Choline Bilayer Membranes . . . . .	283
ROGER E. CRAMER AND RICHARD B. MAYNARD. Calculation of Lanthanide-Induced Shifts from Molecular Structure. III. Other Lanthanides . . . . .	295
ALEX D. BAIN AND JOHN S. MARTIN. Heteronuclear Double Resonance: Individual Overhauser Effects in Coupled $\text{AX}_2$ and $\text{AX}_3$ Spectra . . . . .	301
A. FORMAN AND D. C. BORG. Compensation for Integerization Errors in Simulated EPR Spectra . . . . .	311
<b>COMMUNICATIONS</b>	
C. A. FYFE, J. R. LYERLA, AND C. S. YANNONI. $^{13}\text{C}$ Spectra of Liquid Crystals and Nonrigid Solids Using a High-Resolution Spectrometer . . . . .	315
S. JOHN S. KERRISON AND PETER J. SADLER. The Convention for Referencing Platinum-195 NMR Shifts and the Cooperative Shifts Effect for Chloro-Bromo Complexes of Pt(IV) and Pt(II). . . . .	321
ERIC OLDFIELD AND MICHAEL MEADOWS. Sideways-Spinning 20-mm-Tube Probe for Widebore Superconducting Magnet Spectrometer Systems . . . . .	327
A. G. FERRIGE AND J. C. LINDON. Resolution Enhancement in FT NMR Through the Use of a Double Exponential Function . . . . .	337
I. D. CAMPBELL, C. M. DOBSON, R. G. RATCLIFFE, AND R. J. P. WILLIAMS. A Method for the Accurate Measurement of $^1\text{H}$ Spin-Spin Coupling Constants in Large Molecules . . . . .	341
JAN B. WOOTEN, A. L. BEYERLEIN, JOHN JACOBUS, AND G. B. SAVITSKY. Determination of Deuteron Quadrupole Coupling Constants from Natural Abundance $^{13}\text{C}$ Satellites in $^2\text{H}$ NMR Spectra . . . . .	347
SOOK LEE. Powder Pattern Behavior of the Electron Spin Echo Envelope Modulation Effect . . . . .	351
<b>BOOK REVIEWS</b> . . . . .	
	355

NUMBER 3, SEPTEMBER 1978

F. CARON AND R. F. HERZOG. A New Programmable Timer Designed for Pulsed NMR . . . . .	357
---	-----

K. BARLOS, G. HÜBLER, H. NÖTH, P. WANNINGER, N. WIBERG, AND B. WRACKMEYER. $^{14}\text{N}$ NMR Studies on Some Trimethylsilylamine Derivatives . . . . .	363
M. PUNKKINEN AND L. ÖSTERBERG. Nuclear Dipolar Energy and Relaxation in $(\text{NH}_4)_2\text{GeF}_6$ . . . . .	377
ISAO ANDO AND H. S. GUTOWSKY. Effect of Dielectric Constant upon the Magnetic Anisotropy of the Carbonyl Group and Its Long-Range Chemical Shifts . . . . .	387
D. W. DAVIDSON, S. K. GARG, AND J. A. RIPMEESTER. NMR Behavior of the Clathrate Hydrate of Tetrahydrofuran. II. Deuterium Measurements . . . . .	399
J. F. HARMON. Intermolecular Spin Relaxation by Reorientation of Off-Center Spins . . . . .	411
L. GUIBÉ AND M. C. MONTABONEL. Temperature Dependence of NQR Frequencies in $\text{CuBr}_2$ and $\text{MgBr}_2 \cdot 2\text{Et}_2\text{O}$ . . . . .	419
ASAKO KAWAMORI. Exchange Interaction in Copper Acetate Monohydrate Studied by Nuclear Quadrupole Resonance . . . . .	423
P. W. SMITH AND R. STOESSIGER. Nuclear Quadrupole Resonance Studies of Compounds of Type $\text{A}_2^1\text{M}^{\text{III}}\text{Cl}_5(\text{H}_2\text{O}) \cdot \text{I} \cdot \text{K}_2\text{FeCl}_5 \cdot \text{H}_2\text{O}$ . . . . .	431
TETSUO ASAJI, RYUICHI IKEDA, AND DAIYU NAKAMURA. Anomalous Behavior of the Temperature Dependence of $^{14}\text{N}$ NQR Frequencies in Sodium, Potassium, and Ammonium Thiocyanates . . . . .	437
A. KUMARASWAMY AND J. SOBHANADRI. The EPR of $\text{Gd}^{3+}$ in $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ Single Crystals . . . . .	445
T. E. BULL. Internal Motion in Macromolecules . . . . .	453
GEORGE E. CHAPMAN, BARRY D. ABERCROMBIE, PETER D. CARY, AND E. MORTON BRADBURY. The Measurement of Small Nuclear Overhauser Effects in the $^1\text{H}$ Spectra of Proteins, and Their Application to Lysozyme . . . . .	459
N. S. DALAL, J. A. RIPMEESTER, AND A. H. REDDOCH. $^{14}\text{N}$ NMR Studies of Free Radicals: Application to $\alpha, \alpha$ -Diphenyl- $\beta$ -picrylhydrazyl (DPPH) . . . . .	471
J. R. PILBROW. Effective $g$ Values for $S = \frac{3}{2}$ and $S = \frac{5}{2}$ . . . . .	479
P. A. DE JAGER AND M. A. HEMMINGA. Extension of EPR Spectrometers for the Measurement of Saturation Transfer Spectra . . . . .	491
F. H. A. RUMMENS AND S. RAJAN. The Shielding of a $^3\Sigma$ Hydrogen Gas . . . . .	497

## COMMUNICATIONS

J. C. DUPLAN, A. BRIGUET, G. TETU, AND J. DELMAU. Simultaneous Measurements of the Nuclear Overhauser Effect and $T_1$ . . . . .	509
HAROLD C. NELSON AND JUAN F. VILLA. A Simple Modification to Eliminate Bumping When Using EPR Dewars Containing Liquid Nitrogen . . . . .	515

H. E. BLEICH, K. R. K. EASWARAN, AND JAY A. GLASEL. The Contributions to Amide Proton Spin-Lattice Relaxation in a Small Peptide . . . . .	517
T. OGINO, Y. ARATA, S. FUJIWARA, H. SHOUN, AND T. BEPPU. Use of Proton Correlation NMR Spectroscopy in the Study of Living Cells. Anaerobic Metabolism of <i>Escherichia coli</i> . . . . .	523
ERIC OLDFIELD AND ROBERT P. SKARJUNE. Spin-Echo and Spin-Lock Natural-Abundance Carbon-13 Fourier Transform NMR of Proteins Using a Sideways-Spinning 20-mm Tube Probe . . . . .	527
W. P. AUE AND R. R. ERNST. Scaling of Heteronuclear Spin Coupling by Multipulse Techniques . . . . .	533
ANNOUNCEMENTS AND NEWS ITEMS . . . . .	539
AUTHOR INDEX FOR VOLUME 31 . . . . .	541

The Subject Index for Volume 31 will appear in the December 1978 issue as part of a cumulative index for the year 1978.



## Information for Authors

The *Journal of Magnetic Resonance* includes original papers, both full articles and communications, dealing with the theory, techniques, methods or spectral analysis and interpretation, spectral correlations and results of magnetic resonance spectroscopy and related fields. The Editor seeks the assistance of expert referees in the evaluation of manuscripts of articles, but he alone is responsible for the final decision concerning acceptance.

Original papers only will be considered. Manuscripts are accepted for review with the understanding that the same work has not been and will not be published nor is presently submitted elsewhere, and that all persons listed as authors have given their approval for the submission of the paper; further, that any person cited as a source of personal communications has approved such citation. Written authorization may be required at the Editor's discretion. Articles and any other material published in the *Journal of Magnetic Resonance* represent the opinions of the author(s) and should not be construed to reflect the opinions of the Editor(s) and the Publisher.

Authors submitting a manuscript do so on the understanding that if it is accepted for publication, copyright in the article, including the right to reproduce the article in all forms and media, shall be assigned exclusively to the Publisher. The Publisher will not refuse any reasonable request by the author for permission to reproduce any of his or her contributions to the journal.

Communications are preliminary accounts of work of special importance or contain discussion of controversial subjects. Communications must be especially carefully and concisely prepared; since they may not be refereed, it is the responsibility of the author to ensure that the manuscript as originally submitted is free of typographical or substantive errors. To save time in publication, proofs of communications are not sent to the authors for correction.

Fifty reprints of each paper are supplied without charge, and additional reprints may be ordered on a form which is sent to the author. For articles, the reprint order form accompanies the galley proofs which the author receives. The Journal *assesses no page or publication charges*.

All manuscripts and books for review should be sent to the editor, Wallace S. Brey, Department of Chemistry, University of Florida, Gainesville, Florida 32611.

### Arrangement of the Paper

If the paper is lengthy, it should be divided into sections, although it is preferred that the sections not be numbered. Tables are numbered consecutively with *Arabic* numerals and are to be mentioned in order in the text. Each table should be supplied with a title. Figures are also numbered consecutively with *Arabic* numerals and are mentioned in order in the text. Each regular article requires an abstract which should describe concisely the substantive content, the conclusions reached, and the contributions of the research described. Since the abstract may be used directly by Chemical Abstracts and other abstracting services, it must be self-contained, having no references to formulas, equations, or bibliographic citations which appear in the body of the manuscript.

Literature references are cited in numerical order in the text by *in-line, parenthesized, italic numerals*. References to "unpublished" or "to be published" work from the author's laboratory should not be given. However, dissertations may be cited, or papers actually accepted may be referred to as "in press" if the name of the journal is included.

Authors are urged to give careful thought to the logical construction of the manuscript, so that explanatory or parenthetical footnotes need not be employed—where possible, such materials should be incorporated in the text. Any footnotes which are indispensable are not intermixed with references, but are indicated in the text by consecutive, superscript numerals, and each footnote will then appear in the print at the bottom of the page on which it is cited. Equation numbers are given in *square brackets* to the right of the equation, and references in the text to equations should be in the form "Eq. [3]."

### Form of Manuscript

Manuscripts should be submitted in triplicate. The *original typewritten copy* should be supplied; mimeo, electrostatic and similar process reproductions are not usually sufficiently clear. All of the typing must be

*double-spaced*, including that of abstract, references, and footnotes, on one side of good quality paper approximately 22 × 28 cm (8.5 × 11 in.) in dimensions. The first line of each paragraph is indented.

Each page of the manuscript should be numbered. The first page contains the article title, the author's name, and affiliation. At the bottom of this page should appear any footnotes to the title (indicated by superscript \*, †, ‡); the number of manuscript pages, figures, and tables should also be noted. The second page should contain the abstract of 50–200 words.

*Symbols and Abbreviations.* The American Chemical Society's Handbook for Authors, 1967 edition or the Style Manual of the American Institute of Physics should be followed for standard abbreviations, names and symbols for units. Mathematical equations or symbols must be typewritten whenever possible. Greek letters may be identified in pencil in the margin. The author of any manuscript in which there are mathematical equations or symbols is urged to supply a list of these on a separate sheet for the assistance of the printer in selecting the proper type. The list will not appear in print.

*References and Footnotes.* References to the literature should be cited in order in the text by in-line, parenthesized numerals. The references themselves are to be typed double-spaced on a separate sheet in numerical order. Each reference contains the author's initials, last name, journal name, volume, initial page number and year in parentheses, *in that order*. The name of the journal is abbreviated in the style of Chemical Abstracts' *Service Source Index* (1969 Edition). For book references, the form is author's name, name of the book in quotation marks, editor's name (if any), edition if other than the first, chapter or page number, publisher's name, place of publication, and year of publication.

Any footnotes which are included are numbered in a sequence separate from the references and must be typed in the manuscript on a separate page.

*Tables.* Tables should be laid out carefully, so that minimum space is used and entries are accurately grouped and clearly labeled. Usually, a table should be arranged vertically, with more rows than columns. Vertical lines are not used to separate the columns. Each table is numbered with an Arabic numeral, provided with a title, and typed on a separate sheet of paper. Footnotes to the table are placed directly below it and are indicated by superscript, lower case, italic letters (*a, b, c*). Tables that are longer than two manuscript pages will be reproduced photographically, and should therefore be typed in the exact form desired. All such tables should be carefully checked since errors cannot be corrected in proof.

*Figures.* Figures must be carefully drawn in black, waterproof drawing ink, to draftsman's standards, with lettering by stencil or drawing machine. Freehand, penciled, or typewritten lettering is not acceptable. Lettering should include numerical scales and units required for the two axes, and should be large enough to be legible after reduction by 50–60%. The illustration copy should be on sheets of the same size as that on which the manuscript is typed. Smaller figures may be mounted on sheets of the required size, and larger originals may be handled by supplying glossy, high-contrast, photographic reductions. The original and two duplicates of each figure are required.

*Diagrams.* Ink drawings should be supplied for any complex molecular formulas or diagrams involving material which the printer cannot readily set in type.

*Proofs.* Galley proofs will be sent to the author with reprint forms. Fifty reprints of each article will be supplied free of charge.

Academic Press publishes books and journals in many areas of physics including:

condensed matter

molecular, particle and high energy physics

astronomy and astrophysics

electromagnetism

optics

quantum electronics

mathematical and theoretical physics

atomic, molecular and plasma physics

nuclear engineering

For a list of titles in your subject area, please write to the publisher, attention: Sales Department.

AP 7409

**ACADEMIC PRESS**

*A Subsidiary of Harcourt Brace Jovanovich, Publishers*

111 FIFTH AVENUE, NEW YORK, N.Y. 10003  
24-28 OVAL ROAD, LONDON NW1 7DX

"... an excellent series of collected monographs . . .  
excellent compendium . . . deserves a place in the libraries  
of working physicists."

—PHYSICS TODAY

## ADVANCES IN ATOMIC AND MOLECULAR PHYSICS

Edited by D. R. BATES and BENJAMIN BEDETERSON

### FROM REVIEWS OF PUBLISHED VOLUMES:

"This volume maintains the authoritative standards of the series . . . The editors and publishers are to be congratulated. . . ."

—PHYSICS BULLETIN

". . . maintains the high standards of earlier volumes in the series. . . . All the articles are written by experts in the field, and their summaries are most timely . . . . Strongly recommended to all those interested in collision phenomena and related topics."

—AMERICAN SCIENTIST

"It is very much hoped that the series will . . . find a place alongside its more chemical neighbors in all forward-looking libraries."

—JOURNAL OF THE FARADAY SOCIETY

Complete information on each volume in the series is available on request.

**Take advantage of the convenience of our Continuation Order Plan:**  
Your CONTINUATION ORDER authorizes us to ship and bill each volume automatically, immediately upon publication. This order will remain in effect until cancelled. Please specify volume number with which your order is to begin. Please direct all inquiries and orders to the Sales Department.

AP 7414

# ACADEMIC PRESS

A Subsidiary of Harcourt Brace Jovanovich, Publishers

111 FIFTH AVENUE, NEW YORK, N.Y. 10003  
24-28 OVAL ROAD, LONDON NW1 7DX